

REPORT DOCUMENTATION PAGE			Form Approved OMB No. 0704-0188	
Public reporting burden for this collection of information is estimated to average 1 hour per response, including the time for reviewing instructions, searching existing data sources, gathering and maintaining the data needed, and completing and reviewing the collection of information. Send comments regarding this burden estimate or any other aspect of this collection of information, including suggestions for reducing this burden to Washington Headquarters Services, Directorate for Information Operations and Reports, 1215 Jefferson Davis Highway, Suite 1204, Arlington, VA 22202-4302, and to the Office of Management and Budget Paperwork Reduction Project (0704-0188), Washington, DC 20503.				
1. AGENCY USE ONLY (Leave blank)		2. REPORT DATE January 7, 2004		3. REPORT TYPE AND DATES COVERED Final Technical 6/1/02 - 11/30/03
4. TITLE AND SUBTITLE Very Low Defect Density 4H-SiC (112-0) Thin Films and Their Application to High Power Devices			5. FUNDING NUMBERS	
6. AUTHOR(S) Robert F. Davis				
7. PERFORMING ORGANIZATION NAME(S) AND ADDRESS(ES) North Carolina State University Hillsborough Street Raleigh, NC 27695			8. PERFORMING ORGANIZATION REPORT NUMBER N00014-02-1-0580	
9. SPONSORING/MONITORING AGENCY NAMES(S) AND ADDRESS(ES) Sponsoring: ONR, Code 312, 800 N. Quincy, Arlington, VA 22217-5660 Monitoring: Administrative Contracting Officer, Regional Office Atlanta Suite 4R15 100 Alabama Street, NW Atlanta, GA 30303-3104			10. SPONSORING/MONITORING AGENCY REPORT NUMBER	
11. SUPPLEMENTARY NOTES				
20040209 084				
12a. DISTRIBUTION/AVAILABILITY STATEMENT Approved for Public Release; Distribution Unlimited				
13. ABSTRACT (Maximum 200 words) Investigations have been concerned with substrate etching, thin film growth and doping and the growth of pin structures coupled with structural, microstructural, thickness, optical and electrical characterizations. Maps of the FWHM of the x-ray rocking curves reveal that both the number and the degrees of tilt of the domains in the (112-0) wafers were significantly smaller than those observed for the 8° off-axis (0001) as-received wafers. The average FWHM values for the former and latter were determined to be 22.93 and 50.96 arcsec, respectively. Growth results obtained on 4H-SiC(112-0) have shown differences with those obtained on 8° off-axis 4H-SiC(0001). The film thickness varied from 30 to 80 µm for the samples grown. With Nomarski interference optical microscopy the axis of the micropipes were oriented parallel to the surface in the (112-0) oriented wafers. These defects did not propagate into the epitaxial layer, even if they intersected the initial growth surface. Hall measurements of the [112-0]-oriented samples yielded a bulk mobility of 12,200 and 800 cm ² /Vs at 100°K and 300°K respectively with a carrier concentration (N _p) ranging from 3.1x10 ¹⁴ /cm ³ at 100°K to 7.4x10 ¹⁴ /cm ³ at 300°K. These values met the DARPA deliverable target values for this metric. The results indicate higher electron mobilities than reported previously for 4H-SiC(0001) films.				
14. SUBJECT TERMS SiC, chemical vapor deposition, FWHM, TEM, Hall measurements, Nomarski interference, RMS surface roughness values, PiN material structures			15. NUMBER OF PAGES 13	
			16. PRICE CODE	
17. SECURITY CLASSIFICATION OF REPORT UNCLAS	18. SECURITY CLASSIFICATION OF THIS PAGE UNCLAS	19. SECURITY CLASSIFICATION OF ABSTRACT UNCLAS	20. LIMITATION OF ABSTRACT SAR	

Final Technical Report

Submitted to

Dr. Harry Dietrich
Office of Naval Research
Ballston Tower One
800 North Quincy Avenue
Arlington, VA 22217-5660

For the period

June 1, 2002 – November 30, 2003

For the program

Wide Band Gap Semiconductor Technology Initiative

Program Code: 01-35

ONR Reference No: N00014-02-1-0580

Concerning the Proposal

"Very Low Defect Density 4H-SiC(11 $\bar{2}$ 0) Thin Films and
Their Application to High Power Devices"

P. I.: Robert F. Davis
Department of Materials Science and Engineering,
North Carolina State University
Raleigh, NC 27695-7907
robert_davis@ncsu.edu

DISTRIBUTION STATEMENT A
Approved for Public Release
Distribution Unlimited

07 January 2004

1. EXECUTIVE SUMMARY

The NCSU high temperature SiC growth laboratory and chemical vapor deposition system, including all associated subsystems, have been completed and commissioned. Utilization of the growth system has been delayed because of problems with the induction heating assembly. These equipment development efforts have been conducted simultaneously and in tandem with SiC thin film growth and characterization studies in the laboratory of Professor Janzen at Linköping University in Sweden.

The investigations in Sweden have been concerned with substrate etching, thin film growth and doping and the growth of pin structures coupled with structural, microstructural, thickness, optical and electrical characterization. Maps of the FWHM of the x-ray rocking curves acquired at NCSU from the as-received (1120) wafers revealed that both the number and the degree of tilt of the domains in these substrates were significantly smaller than those observed for the as-received 8° off-axis (0001) wafers. The average FWHM values for the former and latter were determined to be 22.93 and 50.96 arcsec, respectively. The RMS roughness of the (1120) and (0001) as-received wafer surfaces were 0.52 nm and 1.67 nm, respectively. Hydrogen etching of the (1120) surface for five and thirty minutes decreased the as-received RMS roughness to 0.48 nm and 0.28 nm, respectively.

The film thickness varied from 30 to 80 μm for the samples grown. The RMS surface roughness of the epilayers grown on the (1120) substrates after 30-80 μm of growth was 0.10 to 0.60 nm. The micropipes were observed using Nomarski interference optical microscopy to be oriented parallel to the surface of the (1120) wafers and did not propagate into the epitaxial layer. Hall measurements of the [1120]-oriented samples yielded a bulk mobility of 12,200 cm^2/Vs (100°K) and 800 cm^2/Vs (300°K) and with a carrier concentration (N_D) ranging from $3.1 \times 10^{14}/\text{cm}^3$ (100°K) to $7.4 \times 10^{14}/\text{cm}^3$ (300°K). These results indicate higher electron mobilities with than reported previously for 4H-SiC(0001) films. The sharp free exciton line (FE) present in the photoluminescence spectra of a [1120]-oriented film was comparable to that obtained from a high-purity (0001) sample (acquired in the [1120] direction), indicating good sample purity.

Seven PiN structures were also grown in Sweden: five on 4H-SiC(1120) and two on 8° off-axis 4H-SiC(0001). The device layers for all seven PiN samples have similar thicknesses and doping levels; however, the substrates were etched for 15-50 minutes and the buffer layer thicknesses varied from 0-3.6 μm . Selected PiN structures were subsequently fabricated into devices and characterized electrically at Rockwell Scientific. Measurements of 1500 μm and 2000 μm (1120) devices yielded median forward voltages smaller than corresponding (0001) devices. The median blocking voltage for all sizes of the (1120) devices was greater than that measured for equivalent (0001) devices.

The film thickness uniformity across a 4H-SiC(1120) sample was determined at the Naval Research Laboratories (NRL) to be less than 5%, meeting the DARPA deliverable target for this metric. The same sample was analyzed by AFM and yielded RMS surface roughness values of 0.62 – 0.72 nm in agreement with RMS values found at NCSU. Electroluminescence studies were performed at NRL on (1120) and (0001) diodes at

100A/cm². The latter exhibited a 1V increase in forward voltage drop from 9V to 10V after 17 seconds while the former increased 0.04V after 5 minutes.

2. PROGRESS IN STATEMENT OF WORK TASKS (1), (2), (3), AND (4)

2.1 Design and Construction of SiC Thin Film Laboratory and Growth System at NCSU

The construction and upfit of the NCSU laboratory for high-temperature growth of SiC films has been completed. The NCSU chemical vapor deposition system for high-temperature growth of SiC, shown in Figure 1, and associated component systems, including vacuum, RF heating, and gas flow monitoring and control have been established, connected and commissioned. The writing and installation of custom compatible software for automation and control has also been completed. Magnetic cross-talk between the induction coils and the susceptibility of the insulation surrounding the growth chamber (see Problem Areas below) has limited the utilization of the system to this date; however, work is ongoing to solve these problems.

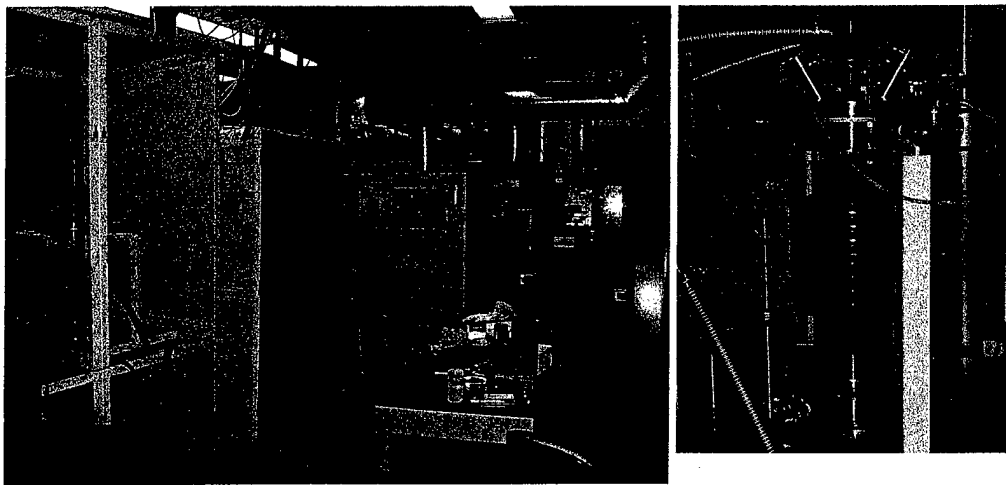


Figure 1. Photographs of the NCSU high-temperature chemical vapor deposition system for the growth of 4H-SiC thick films

2.2 Growth and Characterization of 4H-SiC(11 $\bar{2}$ 0) films at Linköping University NCSU and the Naval Research Laboratory

2.2.1 Experimental Procedures

X-ray diffraction mapping, using a Phillips X'Pert MRD diffractometer, and atomic force microscopy (AFM) with a Digital Nanoscope 3000 in the tapping mode were conducted at NCSU on both 4H-SiC(11 $\bar{2}$ 0) and 8° off-axis 4H-SiC(0001) wafers/substrates to compare and contrast the full-width half-maxima (FWHM) of their rocking curves and their surface morphologies, respectively. The rocking curves were used to characterize the size and distribution of the domains within the SiC wafers. These and additional n-type and p-type substrates were then carried by the P. I. and his students to Professor Janzen's group in Linköping, Sweden to initiate a long-term, on-site collaboration regarding the surface preparation of these substrates and the growth and characterization of SiC thin films and material PiN device structures.

The substrates were subsequently etched in flowing H₂ at 1580°C 5-to-30 minutes in the SiC thin film growth chamber. Six 4H-SiC(11 $\bar{2}$ 0) homoepitaxial films having n-type character and various thicknesses and structures were grown immediately on the etched substrates in an Epigress RF heated, horizontal, hot wall chemical vapor deposition reactor at 1580°C and 200 Torr total pressure. The precursor gases for silicon and carbon were SiH₄ and C₂H₄, respectively. Hydrogen was used both as the carrier gas for SiH₄ and the diluent. Growth rates to 5 μ m/hr were achieved. The information for these samples is summarized in Tables I.

Table I: Sample summary for the initial growth of 4H-SiC(11 $\bar{2}$ 0) films.

Sample ID	Substrate Doping	H-Etch (min)	Layer Structure	Thickness (microns)	Total Thickness (microns)
CV0211B-03	n-type	10	n-	27	27
BQ0255A-41	p-type	10	n-	27	27
CV0211B-95	n-type	30	n-/n-	30/30	60
BQ0255A-83	p-type	5	n-/n+	20/1	21
BQ0255A-88	p-type	30	n-/n-/n-/n+	30/30/20/1	81
CV0211B-53	n-type	30	n-/n-/n-/n+	30/30/20/1	81

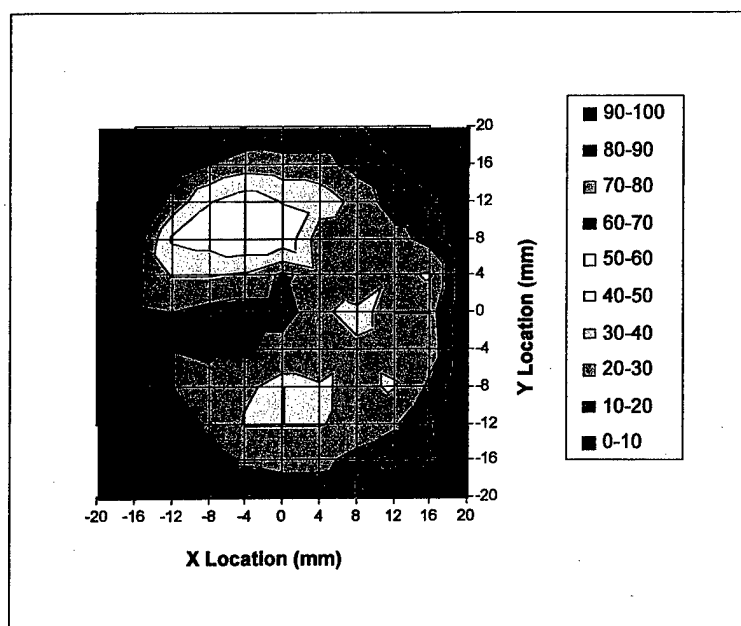
Microstructural characterization of the films was performed in a manner similar to that used to investigate the substrates. Nomarski interference optical microscopy was employed to interrogate the film-substrate interface. Bulk mobilities and carrier concentrations in the (11 $\bar{2}$ 0) films were determined using Hall measurements (Van der Pauw configuration) and compared with analogous published data for 8° off-axis 4H-SiC(0001) materials. Low temperature photoluminescence (LTPL) experiments were performed in a bath cryostat with liquid helium pumped below the lambda point (2K). The excitation source was the 244 nm line of a FreD laser (the double frequency of a 488 nm Ar⁺ ion laser). A single JY-HR460 monochromator fitted with a grating with 2400 groves/mm blazed at 330 nm was used to disperse the luminescence into a UV-sensitive

CCD camera. These measurements were performed in the back scattering geometry, where excitation and detection are perpendicular to the sample surface. Both $(11\bar{2}0)$ and (0001) LTPL spectra were recorded in the $[11\bar{2}0]$ direction.

2.2.2 Results and Discussion

Maps of the FWHM of the x-ray rocking curves shown in Figure 2 and acquired from the as-received wafers reveal that both the number and the degree of tilt of the domains in the $(11\bar{2}0)$ wafers were significantly smaller than those observed for the 8° off-axis (0001) wafers. The average FWHM values for the former and latter were determined to be 22.93 and 50.96 arcsec, respectively.

The RMS roughness values of the $(11\bar{2}0)$ and (0001) surface of the as-received wafers were determined using atomic force microscopy (AFM) and were determined to be 0.52 nm and 1.67 nm, respectively. The micrographs presented in Figure 3 show the $(11\bar{2}0)$ surface of three wafers in this study. Hydrogen etching of the $(11\bar{2}0)$ surface for five minutes decreased the roughness to 0.48 nm RMS. Results after 30 minutes of etching yielded 0.28 nm RMS. Hydrogen etching of the (0001) surface at the temperatures used in this research has been shown by several investigators to produce a step-and-terrace microstructure with a common step-height of one unit cell. By contrast, exposure of the $(11\bar{2}0)$ surface under similar conditions only served to reduce the roughness. It could not be determined if the absence of the step-and-terrace microstructure was the result of very wide steps, very narrow steps or the absence of steps



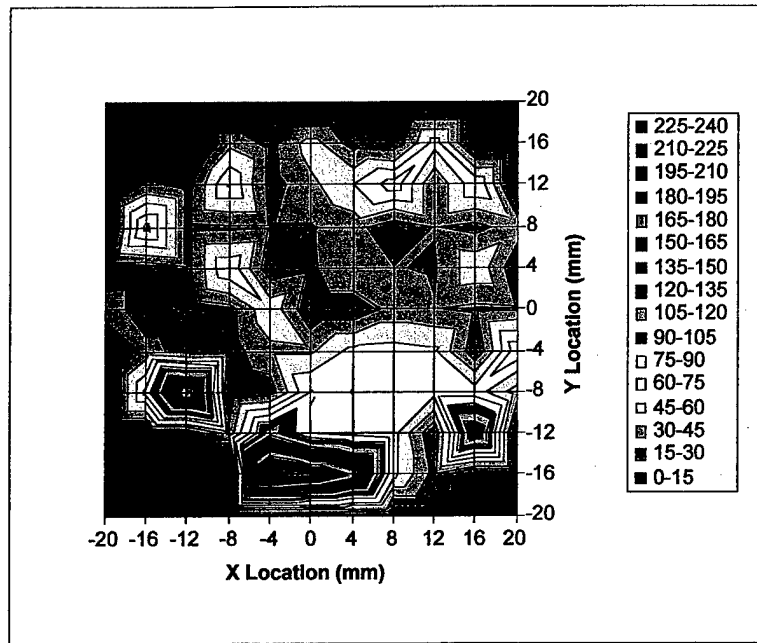


Figure 2. Maps of the FWHM of the x-ray rocking curves of $(11\bar{2}0)$ (top) and 8° off-axis (0001) (bottom) SiC wafers. The average FWHM are 22.93 and 50.96 arcsec, respectively.

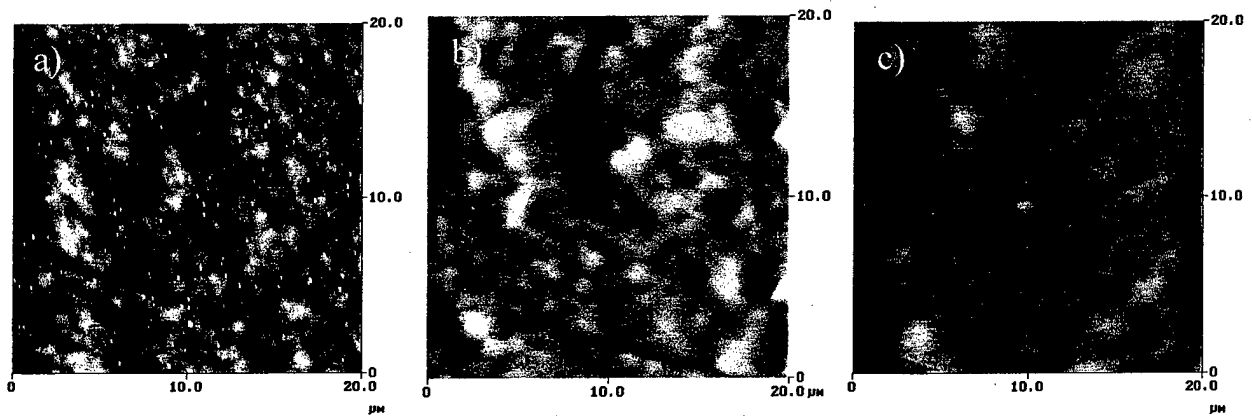


Figure 3. Atomic force microscopy images of 4H-SiC($11\bar{2}0$) wafer surface (a) as-received, and after (b) 5 minutes and (c) 30 minutes of hydrogen etching at 1580°C . Roughness values are 0.52 nm, 0.48 nm, and 0.28 nm RMS, respectively.

Growth results obtained on 4H-SiC($11\bar{2}0$) have shown differences with those obtained on 8° off-axis 4H-SiC(0001). The film thickness varied from 30 to 80 μm for the samples grown.

The RMS surface roughness of the epilayers grown on the ($11\bar{2}0$) substrates after 30-80 μm of growth was determined at NCSU to range from 0.10 to 0.60 nm. Similar studies at NRL on 27 and 80 μm thick epilayers grown in Sweden in this collaborative effort revealed RMS surface roughness values of 0.62 – 0.72 nm, in general agreement with the values determined by the NCSU investigators.

Infrared reflectance thickness studies conducted at NRL on the aforementioned 27 and 81 μm thick epilayers showed the thickness uniformity of the latter sample to be less than 5%, thus meeting the DARPA deliverable target value for this metric. The results of TEM characterization studies at the Army Research Laboratory and of electrical measurements at AFRL and NRL have not been received at this writing.

The results of Nomarski interference optical microscopy are shown and illustrated in Figure 4. The axis of the micropipes were oriented parallel to the surface in the ($11\bar{2}0$) oriented wafers. These defects did not propagate into the epitaxial layer, even if they intersected the initial growth surface.

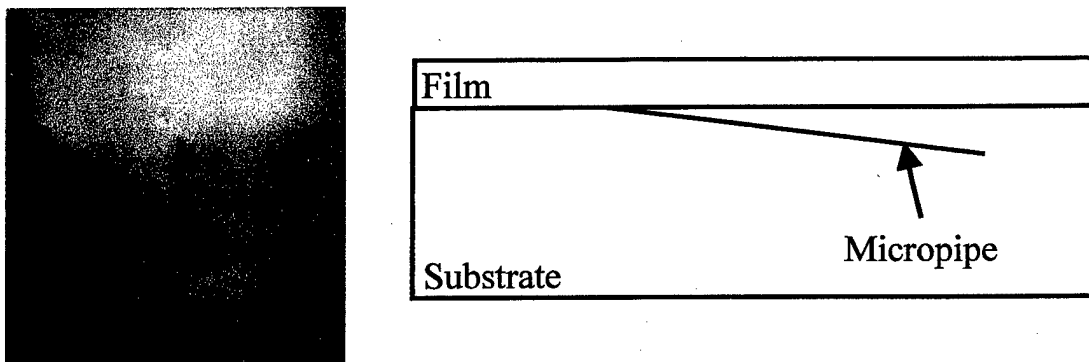


Figure 4. A through focus optical microscopy Nomarski image showing a micropipe that intersected the surface of the substrate prior to growth and but did not propagate into the subsequent homoepitaxial film.

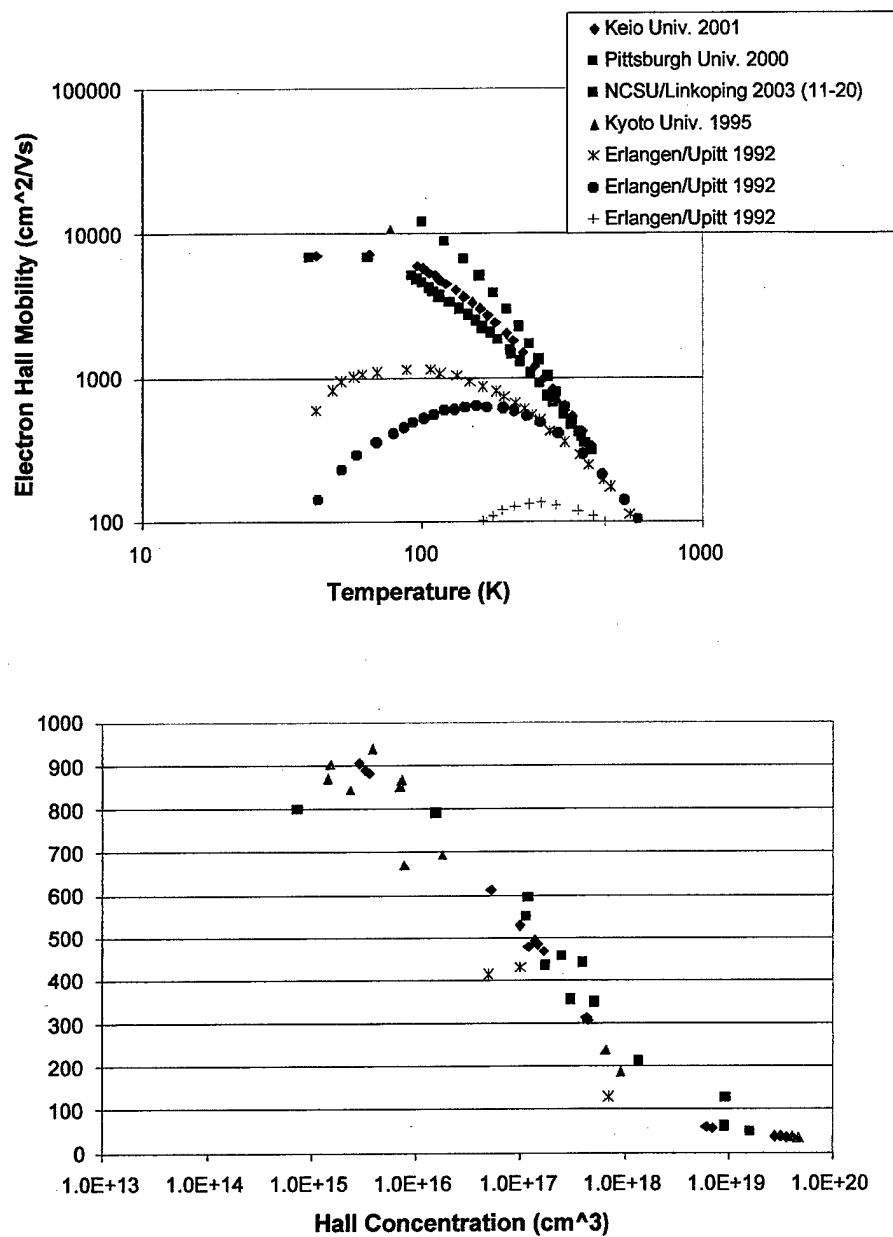


Figure 5. The results of Hall measurements showing bulk mobilities (top) of 12,200 and 800 cm²/Vs at 100°K and 300°K, respectively, and carrier concentrations (N_D) (bottom) ranging from $3.1 \times 10^{14}/\text{cm}^3$ at 100°K to $7.4 \times 10^{14}/\text{cm}^3$ at 300°K.

Hall measurements of the $[11\bar{2}0]$ -oriented samples yielded a bulk mobility of 12,200 and 800 cm²/Vs at 100°K and 300°K respectively with a carrier concentration (N_D) ranging from $3.1 \times 10^{14}/\text{cm}^3$ at 100°K to $7.4 \times 10^{14}/\text{cm}^3$ at 300°K. These values met the DARPA deliverable target values for this metric. As shown in Figure 5, these results indicate higher electron mobilities than reported previously for 4H-SiC(0001) films.

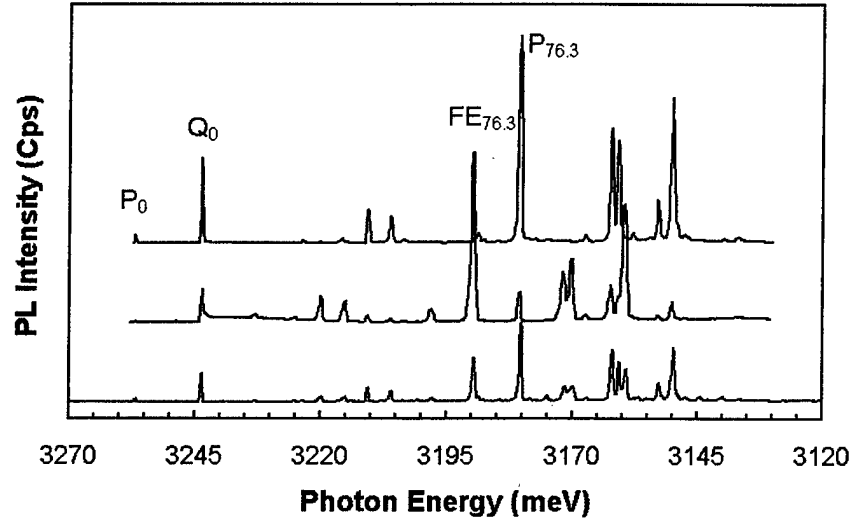


Figure 6. Photoluminescence from the 4H-SiC($11\bar{2}0$) near surface region showing a sharp free exciton (FE) emission line (bottom) as compared to n-doped (0001) sample with a weak FE line (top) and an undoped (0001) sample measured from the a-direction (middle).

The sharp free exciton line (FE) present in the photoluminescence data acquired from the [$11\bar{2}0$] oriented sample and presented in Figure 6 indicates good sample purity. It is comparable to that obtained from a high-purity (0001) sample wherein the PL spectra were acquired in the [$11\bar{2}0$] direction. These data are in contrast to the suppressed free-exciton signature acquired from an n-doped (0001) sample.

3. Fabrication and Characterization of 4H-SiC($11\bar{2}0$)- and (0001)-based High-power PiN Devices

3.1 Experimental Procedure

PiN material structures were grown on n-type 4H-SiC($11\bar{2}0$) and (0001) wafers in collaboration with Professor Janzen's group. The same process parameters used for the chemical vapor deposition parameters of the epilayers were also employed for the growth of the device structures. The characteristics of these assemblies are given in Table II. Selected structures were fabricated into 250 to 2000 μm devices and characterized by Rockwell Scientific. Diode fabrication included JTE implant, field oxide deposition, and a metalization step for frontside and backside ohmic contacts. 200 \AA of aluminum and 1000 \AA of nickel were deposited for the former and 1000 \AA of Ni for the latter. The metal sintering cycle was 1070°C for 10 sec. in Ar.

3.2 Results and Discussion

The results from breakdown voltage and forward voltage measurements performed by Rockwell Scientific are shown in Figure 7 and summarized in Table III. The median forward voltage for the $(11\bar{2}0)$ devices having side lengths of 250 500 and 1000 μm was higher than that for the corresponding (0001) devices. However as the device size increased to 1500 and 2000 μm , the median forward voltage for the $(11\bar{2}0)$ devices became smaller than that for the corresponding (0001) devices. In addition, the median blocking voltage for the $(11\bar{2}0)$ devices of all sizes was greater than those for the equivalent (0001) devices. It should be noted that optimum conditions for the growth of these device structures must still be determined. Moreover, a standard metallization

Table II: Sample summary for the PiN material structures.

Sample ID	Plane	H-Etch (min)	Layer Thickness (microns)				Total Thickness (microns)
			n+ buffer	n- layer	p layer	p+ layer	
BL0221-20	$(11\bar{2}0)$	50	0.3	28.3	1.5	1	31.1
BX0248-30	$(11\bar{2}0)$	15	0.3	26.7	1.7	1	29.7
BX0248-57	$(11\bar{2}0)$	15	3.6	25.5	1.6	0.9	31.6
BX0248-18	$(11\bar{2}0)$	40	0.3	29.5	1.6	0.9	32.3
BX0292-27	$(11\bar{2}0)$	15	--	>30	2.2	1.2	>30
AC0350-05	(0001)	15	0.3	29.1	1.9	1	32.3
AC0113-06	(0001)	15	4	28.4	1.8	1	35.2

Table III – Median forward voltage and median blocking voltage for $(11\bar{2}0)$ and (0001) diodes of varying size

Device Size (side length in μm)	Median Forward Voltage (V)		Median Blocking Voltage (V)	
	$(11\bar{2}0)$	(0001)	$(11\bar{2}0)$	(0001)
250	8.12	4.95	1944	63
500	6.62	4.42	1697	76
1000	4.81	4.24	959	71
1500	4.92	5.15	399	107
2000	3.84	6.13	268	75

procedure must be developed for $[11\bar{2}0]$ -oriented materials and devices, as it was determined in this research that procedures now used for $[0001]$ -oriented devices are not applicable for the $[11\bar{2}0]$ -oriented structures. Capacitance-voltage measurements were also performed by Rockwell Scientific. Values of $N_D - N_A$ of $6.07 \times 10^{14}/\text{cm}^3$ and $5.17 \times 10^{14}/\text{cm}^3$ were determined for the $[11\bar{2}0]$ - and the $[0001]$ -oriented epitaxial layers, respectively.

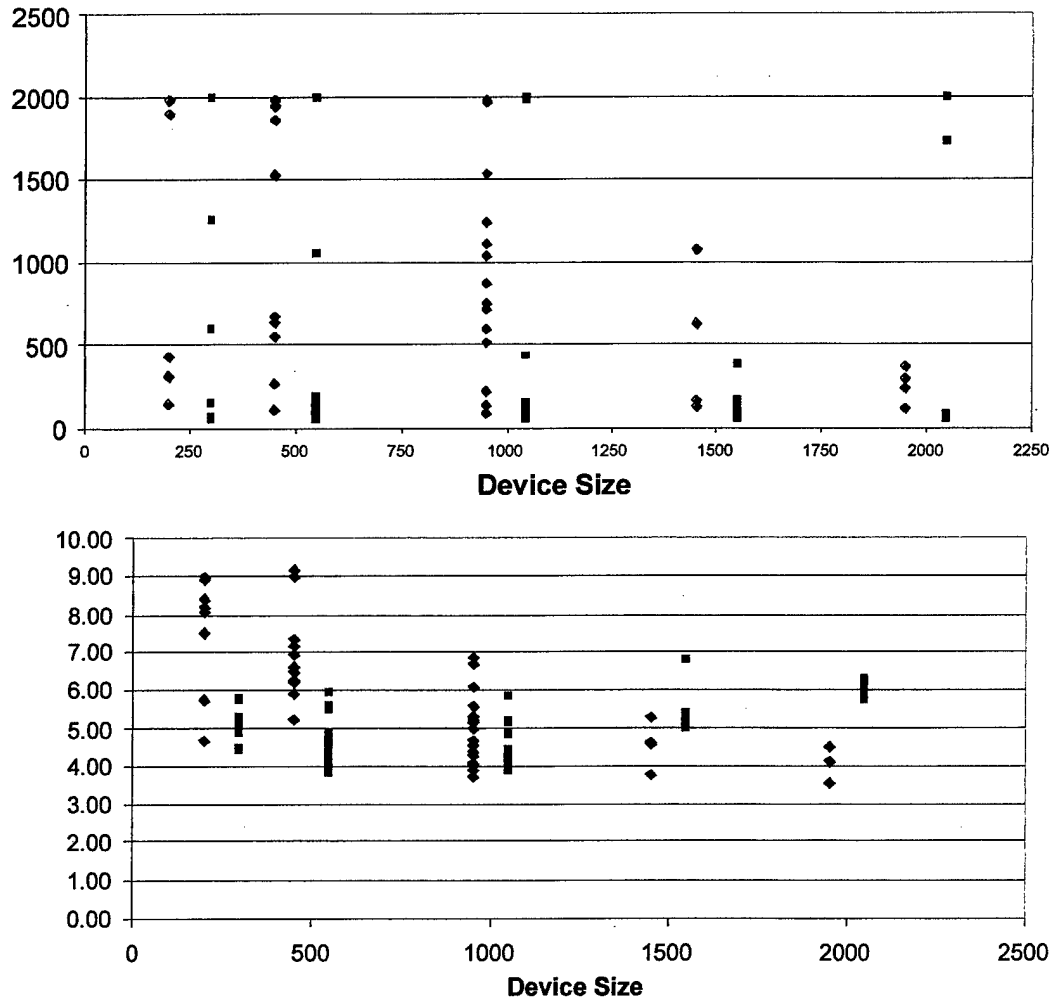


Figure 7. Blocking voltage (top) and forward voltage (bottom) results for 4H-SiC($11\bar{2}0$) (blue) and 4H-SiC(0001) (pink) diodes with side lengths ranging from 250 μm to 2000 μm .

Dr. Robert Stahlbush at NRL performed preliminary electroluminescence studies at $100\text{A}/\text{cm}^2$ on 4H-SiC($11\bar{2}0$) and 4H-SiC(0001) devices selected from those fabricated and characterized by Rockwell Scientific. The ($11\bar{2}0$)-oriented diode exhibited a 1 V increase in the forward voltage drop from 9V to 10V after 17 seconds. Stacking faults

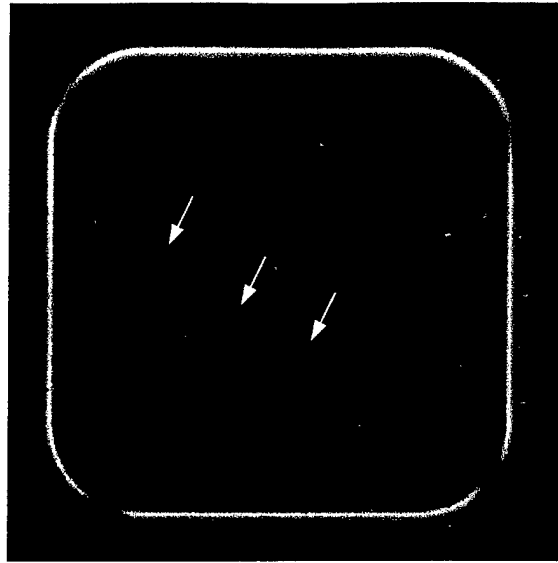


Figure 8. Image of a 4H-SiC($11\bar{2}0$) diode stressed at 100 A/cm^2 for 17 sec. Stacking faults (white arrows) were observed propagating parallel to the surface.

were observed “growing” parallel to the surface, as shown in Figure 8. The forward voltage in the (0001) diode increased 0.04V after 5 minutes of testing.

4. Major Accomplishments:

- Construction and upfit of the NCSU laboratory for high temperature growth of SiC was completed.
- 4H-SiC(11 $\bar{2}$ 0) films to 81 microns thickness were grown on 4H-SiC(11 $\bar{2}$ 0) substrates in Professor Janzen's laboratory.
- The NCSU system for high temperature growth of SiC and associated component systems, including vacuum, RF heating, and gas flow monitoring and control have been established, connected and commissioned.
- The writing and installation of custom compatible software for automation and control was completed.
- Six films of various thickness and structures were grown on the hydrogen-etched n-type and p-type 4H-SiC(11 $\bar{2}$ 0) wafers in the Janzen Laboratories in Linköping, Sweden. PiN device structures were grown on 4H-SiC(11 $\bar{2}$ 0) and 8° off-axis 4H-SiC(0001) substrates.
- The films were characterized via AFM, PL and Hall measurements at NCSU and Linköping University. AFM and Infrared reflectance thickness studies were conducted at NRL. Additional characterization of the films is ongoing at ARL and AFRL.
- Infrared reflectance thickness studies conducted at NRL on a 27 thick epilayer showed the thickness uniformity to be less than 5%, thus meeting the DARPA deliverable target value for this metric.
- Hall measurements conducted at Linköping University yielded bulk higher electron mobilities than reported previously for 4H-SiC(0001) films as well as a carrier concentration (N_D) ranging from $3.1 \times 10^{14}/\text{cm}^3$ at 100°K to $7.4 \times 10^{14}/\text{cm}^3$ at 300°K; the latter values met the DARPA deliverable target values for this metric.
- PiN devices were fabricated from the aforementioned PiN structures and electrically characterized at Rockwell Scientific. These devices were subsequently characterized via electroluminescence at NRL.
- An abstract for ICSCRM 2003 was submitted and accepted, and the paper was given as an oral presentation.

5. Problem Areas:

- Magnetic crosstalk in the dual coil arrangement used to heat our growth chamber.
- The insulation surrounding the graphite susceptor couples to the magnetic field causing it to heat rather than insulate. New insulation is on order to mitigate this problem.